

**WHAT IS CLAIMED IS:**

1. A method for the production of a hindered phenolic alkyl ester compound comprising:

5 a) reacting methyl acrylate with an alkylphenol compound in the presence of a first catalyst to form a methyl ester intermediate compound,

b) reacting an alcohol having at least 2 carbon atoms with the methyl ester intermediate compound in the presence of a second catalyst to form the hindered phenolic alkyl ester compound,

10 c) neutralizing any catalyst residue with an aqueous phosphoric acid to form a precipitated phosphate salt, and

d) separating the precipitated phosphate salt from the hindered phenolic alkyl ester compound.

15 2. The method according to claim 1, wherein the first and second catalysts have the same chemical composition.

3. The method according to claim 1, wherein the first and second catalysts have different chemical compositions.

20 4. The method according to claim 1, wherein the first catalyst is also used as the second catalyst during the reaction of the alcohol with the methyl ester intermediate compound.

25 5. The method according to claim 1, wherein the first and second catalysts comprise at least one compound selected from the group consisting of alkali and alkaline earth metal hydroxides and oxides, alkali metal hydrides, alkali metal alkoxides, alkali metal amides, zinc salts, calcium salts, monoalkyltins, alkali metal hydrocarbyloxides, and mixtures thereof.

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6. The method according to claim 1, wherein the first and second catalysts comprise at least one compound selected from the group

consisting of potassium hydroxide, sodium hydroxide, lithium hydroxide, cesium hydroxide, calcium hydroxide, magnesium hydroxide, dibutyltin oxide, and mixtures thereof.

5           7.     The method according to claim 1 wherein filtration is used to separate the precipitated phosphate salt from the hindered phenolic alkyl ester compound.

10           8.     The method according to claim 1, wherein the hindered phenolic alkyl ester compound is formed in a substantially liquid form.

          9.     The method according to claim 1, wherein the alkylphenol compound is 2,6-di-tert-butylphenol.

15           10.    The method according to claim 1, wherein the hindered phenolic alkyl ester compound formed is 3,5-di-tert-butyl-4-hydroxyhydrocinnamic acid, alkyl ester.

20           11.    The method according to claim 1, wherein the molar ratio of methyl acrylate to alkylphenol is at least about 1:1.

          12.    The method according to claim 1, wherein a molar excess of methyl acrylate is employed so that the molar ratio of methyl acrylate to alkylphenol is greater than 1:1.

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          13.    The method according to claim 1, wherein a molar excess of methyl acrylate is employed so that the molar ratio of methyl acrylate to alkylphenol is between about 1.05:1 to about 1.30:1.

30           14.    The method according to claim 1, wherein a molar excess of alkylphenol is employed so that the molar ratio of alkylphenol to methyl acrylate is greater than 1:1.

15. The method according to claim 1, wherein a molar excess of alkylphenol is employed so that the molar ratio of alkylphenol to methyl acrylate is between about 1.05:1 to about 1.30:1.

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16. The method according to claim 1, wherein the phosphoric acid comprises at least one compound selected from the group consisting of orthophosphoric acid ( $\text{H}_3\text{PO}_4$ ), pyrophosphoric acid ( $\text{H}_4\text{P}_2\text{O}_7$ ), metaphosphoric acid ( $\text{HPO}_3$ ), phosphorous acid ( $\text{H}_3\text{PO}_3$ ), and  $\text{H}_2\text{PO}_4$ , and mixtures thereof.

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17. The method according to claim 1, wherein the molar equivalent of aqueous phosphoric acid used is between about 10% and 200% of the total amount of catalysts used.

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18. The method according to claim 1, wherein the molar equivalent of aqueous phosphoric acid used is between about 33% and 100% of the total amount of catalysts used.

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19. The method according to claim 1, wherein the alcohol is a high molecular weight alcohol.

20. The method according to claim 1, wherein the methyl acrylate is reacted with the alkylphenol compound in the presence of a promoter.

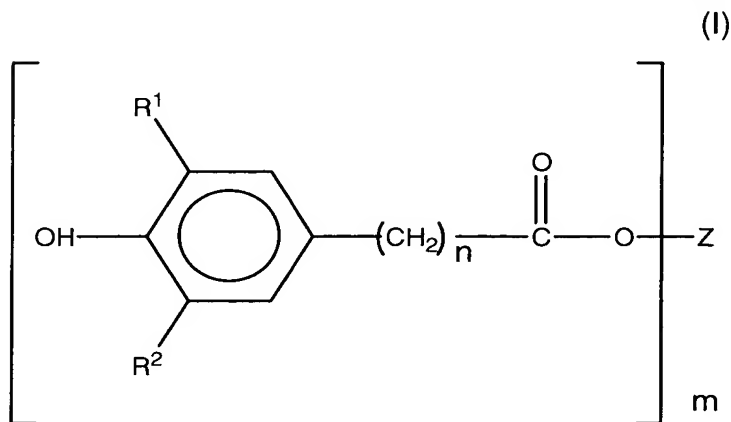
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21. The method according to claim 20, wherein the promoter comprises at least one compound selected from the group consisting of dialkyl sulfoxides, dialkyl formamides, dialkyl ethers, dimethyl acetamide, N,N-dialkyl acidamide, methyl ethyl ketone, methyl butyl ketone, phase transfer agents, crown ethers, and mixtures thereof.

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22. The method according to claim 20, wherein the promoter is tetrahydrofuran.

23. A method for the production of a hindered phenolic alkyl ester compound having the structure according to Formula I:



wherein:

R<sup>1</sup> and R<sup>2</sup> are independently selected from the group consisting of H, alkyl, cycloalkyl, aryl, alkylaryl, and arylalkyl;

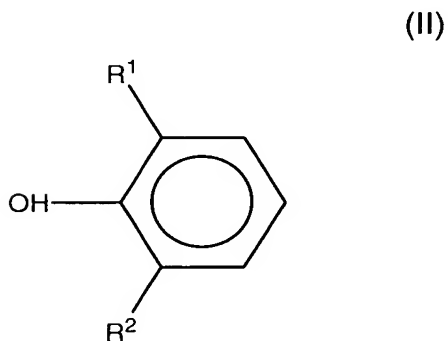
n is 2;

m is 1, 2, 3, or 4; and

Z is alkyl;

comprising the steps of:

a) reacting methyl acrylate with an alkylphenol compound in the presence of a first catalyst to form a methyl ester intermediate compound, wherein the alkylphenol compound has the structure according to Formula II:



and wherein  $R^1$  and  $R^2$  are defined as above;

b) reacting an alcohol having at least 2 carbon atoms with the methyl ester intermediate compound in the presence of a second catalyst to form the hindered phenolic alkyl ester compound having the structure according to Formula I, and

c) neutralizing the catalyst residue with an aqueous phosphoric acid to form a precipitated phosphate salt, and

d) separating the precipitated phosphate salt from the hindered phenolic alkyl ester compound.

24. The method according to claim 23, wherein  $R^1$  and  $R^2$  are alkyl.

25. The method according to claim 23, wherein  $R^1$  and  $R^2$  are independently selected from t-butyl and  $C_1$ - $C_6$  alkyl.

26. The method according to claim 23, wherein  $R^1$  and  $R^2$  are t-butyl.

27. The method according to claim 23, wherein Z is  $C_2$ - $C_{20}$  alkyl.

28. A method for the production of a hindered phenolic alkyl ester compound comprising:

a) reacting methyl acrylate with an alkylphenol compound in the presence of a first catalyst to form a methyl ester intermediate compound,

b) reacting an alcohol having at least 2 carbon atoms with the methyl ester intermediate compound in the presence of a second catalyst to form the hindered phenolic alkyl ester compound, wherein the second catalyst has the same chemical composition as the first catalyst,

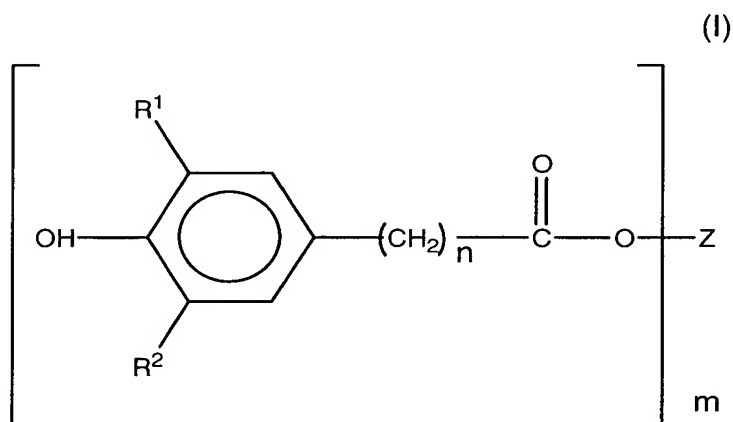
c) neutralizing the first and second catalysts with an aqueous acid to form a precipitated salt, and

d) separating the precipitated salt from the hindered phenolic alkyl ester compound.

29. The method according to claim 28, wherein the first catalyst is also used as the second catalyst during the reaction of the alcohol with the methyl ester intermediate compound.

30. The method according to claim 28, wherein the aqueous acid comprises at least one compound selected from the group consisting of sulfuric acid, nitric acid, hydrobromic acid, hydroiodic acid, hydrochloric acid, formic acid, acetic acid, phosphoric acid, and mixtures thereof.

31. A method for the production of a hindered phenolic alkyl ester compound having the structure according to Formula I:



wherein:

R<sup>1</sup> and R<sup>2</sup> are independently selected from the group consisting of H, alkyl, cycloalkyl, aryl, alkylaryl, and arylalkyl;

n is 2;

m is 1, 2, 3, or 4; and

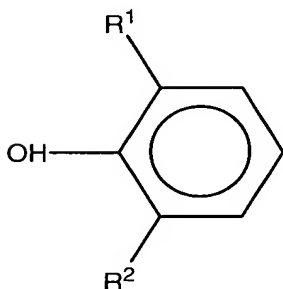
Z is alkyl;

comprising the steps of:

a) reacting methyl acrylate with an alkylphenol compound in the presence of a first catalyst to form a methyl ester intermediate compound, wherein the alkylphenol compound has the structure according to Formula II:

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(II)



and wherein R¹ and R² are defined as above;

b) reacting an alcohol having at least 2 carbon atoms with the methyl ester intermediate compound in the presence of a second catalyst to form the hindered phenolic alkyl ester compound having the structure according to Formula I, wherein the second catalyst has the same chemical composition as the first catalyst,

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c) neutralizing the first and second catalysts with an aqueous acid to form a precipitated salt, and

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d) separating the precipitated salt from the hindered phenolic alkyl ester compound.

32. A method for the production of a hindered phenolic alkyl ester compound comprising:

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a) reacting methyl acrylate with an alkylphenol compound in the presence of a promoter and a first catalyst to form a methyl ester intermediate compound,

b) reacting an alcohol having at least 2 carbon atoms with the methyl ester intermediate compound in the presence of a second catalyst to form the hindered phenolic alkyl ester compound, wherein the hindered phenolic alkyl ester compound is formed in a substantially liquid form,

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c) neutralizing any catalyst residue with an aqueous acid to form a precipitated salt, and

d) separating the precipitated salt from the hindered phenolic alkyl ester compound.

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33. The method according to claim 32, wherein the promoter comprises at least one compound selected from the group consisting of dialkyl sulfoxides, dialkyl formamides, dialkyl ethers, dimethyl acetamide, N,N-dialkyl acidamide, methyl ethyl ketone, methyl butyl ketone, phase transfer agents, crown ethers, and mixtures thereof.

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34. The method according to claim 32, wherein the promoter comprises at least one compound selected from the group consisting of dimethyl sulfoxide, dimethyl formamide, diethyl ether, diisopropyl ether, and mixtures thereof.

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35. The method according to claim 32, wherein the promoter is tetrahydrofuran.

36. A method for the production of a hindered phenolic alkyl ester compound comprising:

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a) reacting methyl acrylate with an alkylphenol compound in the presence of tetrahydrofuran and a first catalyst to form a methyl ester intermediate compound,

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b) reacting an alcohol having at least 2 carbon atoms with the methyl ester intermediate compound in the presence of a second catalyst to form the hindered phenolic alkyl ester compound,

c) neutralizing any catalyst residue with an aqueous acid to form a precipitated salt, and

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d) separating the precipitated salt from the hindered phenolic alkyl ester compound.



37. The method according to claim 36, wherein the alcohol is reacted with the methyl ester intermediate compound in the presence of tetrahydrofuran.

5           38. A method for the production of a hindered phenolic alkyl ester compound comprising:

a) reacting an alkyl acrylate with an alkylphenol compound in the presence of a catalyst to form the hindered phenolic alkyl ester compound,

b) neutralizing the catalyst with an aqueous acid to form a  
10 precipitated salt, and

c) separating the precipitated salt from the hindered phenolic alkyl ester compound.

15           39. The method according to claim 38, wherein the alkyl acrylate is methyl acrylate.

40. The method according to claim 38, wherein the alkyl acrylate is a high molecular weight alkyl acrylate.

20           41. The method according to claim 40, wherein the high molecular weight alkyl acrylate is a C<sub>2</sub>-C<sub>10</sub> alkyl acrylate.

25           42. The method according to claim 41, wherein the C<sub>2</sub>-C<sub>10</sub> alkyl acrylate is a compound selected from the group consisting of n-butyl acrylate, sec-butyl acrylate, n-octyl acrylate, 2-ethylhexyl acrylate, isoheptyl acrylate, isooctyl acrylate, isononyl acrylate, isodecyl acrylate, and mixtures thereof.

30           43. The method according to claim 38, wherein the hindered phenolic alkyl ester compound is formed in a substantially liquid form.

44. The method according to claim 38, wherein the alkyl acrylate is reacted with the alkylphenol compound in the presence of an alcohol.

5 45. The method according to claim 44, wherein the alcohol is a high molecular weight alcohol.

46. The method according to claim 38, wherein the alkyl acrylate is reacted with the alkylphenol compound in the presence of a promoter.

10 47. The method according to claim 46, wherein the promoter comprises at least one compound selected from the group consisting of dialkyl sulfoxides, dialkyl formamides, dialkyl ethers, dimethyl acetamide, N,N-dialkyl acidamide, methyl ethyl ketone, methyl butyl ketone, phase transfer agents, crown ethers, and mixtures thereof.

15 48. The method according to claim 46, wherein the promoter is tetrahydrofuran.

20 49. The method according to claim 38, wherein the aqueous acid comprises aqueous phosphoric acid.

50. A composition produced according to the method of claim 1.

51. A composition comprising:

25 (a) 3,5-di-tert-butyl-4-hydroxyhydrocinnamic acid, C<sub>4</sub>-C<sub>10</sub> alkyl ester, wherein the 3,5-di-tert-butyl-4-hydroxyhydrocinnamic acid, C<sub>4</sub>-C<sub>10</sub> alkyl ester has a gas chromatogram within the composition of about 80.0% to about 98.0% by area,

30 (b) pentanedioic acid, 2-[[3,5-di-tert-butyl-4-hydroxyphenyl]methyl] C<sub>1</sub>-C<sub>10</sub> dialkyl ester, wherein the pentanedioic acid, 2-[[3,5-di-tert-butyl-4-hydroxyphenyl]methyl] C<sub>1</sub>-C<sub>10</sub> dialkyl ester has a gas chromatogram within the composition of about 1.0% to about 20.0% by area, and

(c) 3,5-di-tert-butyl-4-hydroxyhydrocinnamic acid, methyl ester, wherein the 3,5-di-tert-butyl-4-hydroxyhydrocinnamic acid, methyl ester has a gas chromatogram within the composition of about 0.1% to about 5.0% by area.

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52. The composition of claim 51, wherein the 3,5-di-tert-butyl-4-hydroxyhydrocinnamic acid, methyl ester has a gas chromatogram within the composition of about 0.1% to about 1.0% by area.

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53. The composition of claim 51, further comprising 2,6-di-tert-butylphenol, wherein the 2,6-di-tert-butylphenol has a gas chromatogram within the composition of about 0.1% to about 5.0% by area.

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54. The composition of claim 53, wherein the 2,6-di-tert-butylphenol has a gas chromatogram within the composition of about 0.1% to about 1.0% by area.

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55. The composition of claim 51, wherein the 3,5-di-tert-butyl-4-hydroxyhydrocinnamic acid, C<sub>4</sub>-C<sub>10</sub> alkyl ester has a gas chromatogram within the composition of about 90.0% to about 97.0% by area,

the pentanedioic acid, 2-[[3,5-di-tert-butyl-4-hydroxyphenyl]methyl] C<sub>1</sub>-C<sub>10</sub> dialkyl ester has a gas chromatogram within the composition of about 1.5% to about 10.0% by area, and

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the 3,5-di-tert-butyl-4-hydroxyhydrocinnamic acid, methyl ester has a gas chromatogram within the composition of about 0.1% to about 1.0% by area.

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56. The composition of claim 51, wherein:  
the 3,5-di-tert-butyl-4-hydroxyhydrocinnamic acid, C<sub>4</sub>-C<sub>10</sub> alkyl ester has a gas chromatogram within the composition of about 95.0% to about 98.0% by area,

the pentanedioic acid, 2-[[3,5-di-tert-butyl-4-hydroxyphenyl]methyl]  
C<sub>1</sub>-C<sub>10</sub> dialkyl ester has a gas chromatogram within the composition of  
about 1.0% to about 2.5% by area, and

5 the 3,5-di-tert-butyl-4-hydroxyhydrocinnamic acid, methyl ester has  
a gas chromatogram within the composition of about 0.5% to about 1.0%  
by area.

57. The composition of claim 51, wherein:

10 the 3,5-di-tert-butyl-4-hydroxyhydrocinnamic acid, C<sub>4</sub>-C<sub>10</sub> alkyl ester  
is 3,5-di-tert-butyl-4-hydroxyhydrocinnamic acid, isooctyl ester,

the pentanedioic acid, 2-[[3,5-di-tert-butyl-4-hydroxyphenyl]methyl]  
C<sub>1</sub>-C<sub>10</sub> dialkyl ester comprises pentanedioic acid, 2-[[3,5-di-tert-butyl-4-  
hydroxyphenyl]methyl] diisooctyl ester and pentanedioic acid, 2-[[3,5-di-  
tert-butyl-4-hydroxyphenyl]methyl] methyl isooctyl ester.

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58. The composition of claim 51, wherein one of the alkyl ester  
groups of the pentanedioic acid, 2-[[3,5-di-tert-butyl-4-  
hydroxyphenyl]methyl] C<sub>1</sub>-C<sub>10</sub> dialkyl ester is a C<sub>4</sub>-C<sub>10</sub> alkyl ester.

20 59. The composition of claim 51, wherein the pentanedioic acid,  
2-[[3,5-di-tert-butyl-4-hydroxyphenyl]methyl] C<sub>1</sub>-C<sub>10</sub> dialkyl ester comprises  
at least one compound selected from the group consisting of:

pentanedioic acid, 2-[[3,5-di-tert-butyl-4-hydroxyphenyl]methyl]  
dibutyl ester, pentanedioic acid, 2-[[3,5-di-tert-butyl-4-  
25 hydroxyphenyl]methyl] bis(2-ethylhexyl) ester, pentanedioic acid, 2-[[3,5-  
di-tert-butyl-4-hydroxyphenyl]methyl] diisooctyl ester, and pentanedioic  
acid, 2-[[3,5-di-tert-butyl-4-hydroxyphenyl]methyl] diisoheptyl ester, and  
mixtures thereof, and

at least one compound selected from the group consisting of:  
30 pentanedioic acid, 2-[[3,5-di-tert-butyl-4-hydroxyphenyl]methyl] methyl  
butyl ester, pentanedioic acid, 2-[[3,5-di-tert-butyl-4-hydroxyphenyl]methyl]  
methyl 2-ethylhexyl ester, pentanedioic acid, 2-[[3,5-di-tert-butyl-4-

hydroxyphenyl]methyl] methyl isooctyl ester, pentanedioic acid, 2-[[3,5-di-tert-butyl-4-hydroxyphenyl]methyl] methyl isoheptyl ester, and mixtures thereof.

5           60.    A product comprising the composition of claim 51 and at least one material selected from the group consisting of a lubricant oil, a dispersant, a detergent, an antiwear additive, a supplemental antioxidant, zinc dialkyldithiophosphate, an alkylated diphenylamine, a viscosity index improver, a pour point depressant, a corrosion inhibitor, a rust inhibitor, a  
10   foam inhibitor, a supplemental friction modifier, and mixtures thereof.

          61.    A product comprising the composition of claim 51 and at least one material selected from the group consisting of a lubricating oil, a detergent, a dispersant, zinc dialkyldithiophosphate, a corrosion inhibitor, a  
15   rust inhibitor, an alkylated diphenylamine, and mixtures thereof.

          62.    A product comprising the composition of claim 51, a detergent, a dispersant, and zinc dialkyldithiophosphate.

20           63.    The product of claim 62 further comprising a lubricating oil.

          64.    A product comprising the composition of claim 51, a corrosion inhibitor, a rust inhibitor, and an alkylated diphenylamine.

25           65.    The product of claim 64 further comprising a lubricating oil.

          66.    The product of claim 61, wherein the detergent is present in an amount of about 1.0% to about 7.5% by weight of the product, the dispersant is present in an amount of about 1.0% to about 7.5% by weight  
30   of the product, the zinc dialkyldithiophosphate is present in an amount of about 0.5% to about 1.5% by weight of the product, and the composition of

claim 52 is present in an amount of about 0.1% to about 2.0% by weight of the product.

5           67.    The product of claim 64, wherein the corrosion inhibitor is present in an amount of about 0.01% to about 0.5% by weight of the product, the rust inhibitor is present in an amount of about 0.01% to about 0.5% by weight of the product, the alkylated diphenylamine is present in an amount of about 0.1% to about 1.0% by weight of the product, and the composition of claim 52 is present in an amount of about 0.1% to about 10   1.0% by weight of the product.

          68.    The product of claim 60, wherein the lubricant oil is selected from the group consisting of passenger car engine oils, heavy duty diesel engine oils, railroad oils, natural gas engine oils, turbine oils, rust oils, 15   oxidation oils, slideway oils, hydraulic oils, industrial oils, automotive gear oils, automatic transmission fluids and manual transmission fluids, tractor fluids, universal tractor fluids, power steering fluids, gear lubricants, industrial oils, pump oils, and mixtures thereof.

20           69.    A method for the production of a hindered phenolic alkyl ester compound consisting essentially of:  
          a) reacting an alkyl acrylate with an alkylphenol compound in the presence of a catalyst to form the hindered phenolic alkyl ester compound,  
          b) neutralizing the catalyst with an aqueous acid to form a 25   precipitated salt, and  
          c) separating the precipitated salt from the hindered phenolic alkyl ester compound.